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LATHROP & GAGE LC
2345 GRAND AVENUE
SUITE 2800
KANSAS CITY, MO 64108

EXAMINER

SASTRI, SATYA B

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| ART UNIT | PAPER NUMBER |
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1713

DATE MAILED: 07/14/2006

Please find below and/or attached an Office communication concerning this application or proceeding.

Office Action Summary

Application No.

10/671,095

Applicant(s)

GUO ET AL.

Examiner

Satya B. Sastri

Art Unit

1713

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☒ Responsive to communication(s) filed on 15 May 2006.
- 2a) ☒ This action is **FINAL**. 2b) ☐ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 1-9 and 11-78 is/are pending in the application.
- 4a) Of the above claim(s) 19-21, 24, 27-43, 45, 52-54, 56-58, 60, 66-68, 70-72, 74 is/are withdrawn from consideration.
- 5) ☐ Claim(s) _____ is/are allowed.
- 6) ☒ Claim(s) 1-9, 11-18, 22, 23, 25, 26, 44, 46-51, 55, 59, 61-65, 69, 73 and 75-78 is/are rejected.
- 7) ☐ Claim(s) _____ is/are objected to.
- 8) ☒ Claim(s) 1-9, 11-78 are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on _____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some * c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
 2. ☐ Certified copies of the priority documents have been received in Application No. _____.
 3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) ☐ Notice of References Cited (PTO-892)
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) ☐ Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08)
Paper No(s)/Mail Date _____.
- 4) ☐ Interview Summary (PTO-413)
Paper No(s)/Mail Date. _____.
- 5) ☐ Notice of Informal Patent Application (PTO-152)
- 6) ☒ Other: Translation JP 59,179,676.

DETAILED ACTION

1. This office action is in response to amendment filed on May 17, 2006. *Claims 1-9, 11-78* are now pending in the application. Of these, *claims 19-21, 24, 27-43, 45, 52-54, 56-58, 60, 66-68, 70-72, 74* are withdrawn from consideration as non-elected invention.

2. Applicant's amendment and arguments have been fully considered with the following results: Rejection of *claims 50, 51, 59, 64* under 35 U.S.C. 102(b) as anticipated by Kawabata et al. (US 5,620,796), rejection of *claims 1-9, 11-18, 44, 49* under 35 U.S.C. 102(b) as anticipated by or, in the alternative, under 35 U.S.C. 103(a) as obvious over Kawabata et al. (US 5,620,796), rejection of *claims 65 and 73* under 35 U.S.C. 103(a) as being unpatentable over Kawabata et al. (US 5,620,796), rejection of *claims 22, 23, 25, 26, 55, 69* under 35 U.S.C. 103(a) as being unpatentable over Kawabata et al. (US 5,620,796) in view of Phan et al. (US 5,969,032) and rejection of *claims 46-48, 61-63, 75-77* under 35 U.S.C. 103(a) as being unpatentable over Kawabata et al. (US 5,620,796) in view of Gutman et al. (US 5,508,107) are all withdrawn.

However, rejection of *claims 50, 51, 59, 61-63* under 35 U.S.C. 102(b) as anticipated by Narimatsu et al. (EP 0530729 A1), rejection of *claims 1-6, 11-18, 44, 46-49* under 35 U.S.C. 102(b) as anticipated by or, in the alternative, under 35 U.S.C. 103(a) as obvious over Narimatsu et al. (EP 0530729 A1), rejection of *claims 1-3, 6-9, 11-16, 44, 49, 50, 59* under 35 U.S.C. 102(b) as being anticipated by JP 59,179,676 ('676, chemical abstract), rejection of *claims 7-9, 65, 73, 75-78* under 35 U.S.C. 103(a) as being unpatentable over Narimatsu et al. (EP 0530729 A1) and rejection of *claims 4, 5, 65, 73, 78* under 35 U.S.C. 103(a) as being

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unpatentable over JP 59,179,676 ('676, chemical abstract) as set forth in the previous office actions are all sustained.

3. New grounds of rejection are necessitated by the amendment and therefore, this action is made final.

Previously Cited Statutes

4. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

5. **Claim 64** is rejected under 35 U.S.C. 102(b) as anticipated by Narimatsu et al. (EP 0530729 A1).

Prior art teaches pressure sensitive adhesive obtained by emulsion-polymerizing a monomer mixture comprising alkyl (meth)acrylate monomer and 0.1-10 parts by wt. per 100 parts of monomer mixture, of carboxyl group-containing monomer (page 5). (Meth)acrylate monomers are disclosed in lines 4-9, page 5 while anionic monomers are listed in lines 10-11. Aziridine type crosslinking agents in amounts of 0.01 to 10 parts by wt. are disclosed (abstract, page 5, lines 49-51). Working example 1 on page 8 discloses a copolymer of 23 parts of methylmethacrylate, 73 parts of 2-ethylhexyl acrylate, 2 parts of glycidyl acrylate and 2 parts of acrylic acid (page 8, lines 13-16).). 0.5 part by wt. of polyfunctional aziridinyl propionate is employed in this example. In light of above, instant claim is anticipated by the prior art.

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6. *Claims 22, 23, 25, 26, 55, 69* are rejected under 35 U.S.C. 103(a) as being unpatentable over Narimatsu et al. (EP 0530729 A1) in view of Phan et al. (US 5,969,032).

Prior art to Narimatsu et al. is presented above in paragraph 6 and is incorporated herein by reference.

The difference between the prior art and the present invention is that the prior art does not teach the specific polymerizable surfactant in the adhesive composition as claimed instantly.

The primary reference discloses the use of non-polymerizable surfactants. Secondary reference to Phan et al. is in an analogous field of art and discloses the advantages of reactive surfactants for use in preparing acrylic adhesives as opposed to conventional surfactants used compositions disclosed in the primary reference (column 1, lines 43-63). Reactive surfactant such as an allyl amine salt of alkyl benzene sulfonate with allyl amine salt of dodecylbenzene sulfonate is disclosed as the preferred species for polymerization of acrylic latexes (column 5, lines 14-29). The use of such surfactant allows lower levels of polymerizable surfactants to control the latex particle size and to stabilize the latex particles (abstract). In light of such benefits, it would have been obvious for one of ordinary skill in the art at the time the invention was made to include with allyl amine salt of dodecylbenzene sulfonate as the preferred species of surfactant for polymerization of acrylic latexes of Narimatsu et al. and thereby obtain the present invention.

Response to Arguments

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7. With regard to prior art to Narimatsu et al., applicants contend that it does not teach or suggest the instant invention. Applicants' attention is drawn to working example 1 that includes an aqueous emulsion of a hydrophobic monomer (73 parts of 2ethylhexyl acrylate, hydrophilic monomer (2 parts of methacrylic acid), and a partially hydrophilic monomer (23 parts of methyl methacrylate). To 100 parts by wt. of solid content, 0.5 part of tetramethylol tri- β -aziridinypropionate is added as crosslinking agent. Thus, the composition is explicitly taught by the prior art. The initial adhesion strength on a corona discharged EVA and PP films is 180 g/25 mm. Although the substrates are different, since the prior art concerns adhesives useful for good peelability (abstract), a reasonable basis exists to believe that the prior art compositions would inherently possess an adhesion strength of less than 2 lbs/inch when used on polyester films as recited in instant claims.

8. Similarly, JP 59,179,676 clearly discloses polyfunctional aziridine propionate as crosslinking agent (page 12, paragraphs 2-3). Thus, applicants' arguments that the prior art does not teach or suggest polyfunctional aziridine propionate as crosslinking agent are not found persuasive. A complete English translation of JP 59179676 used as prior art in previous rejections is being attached herewith.

Action Is Final

9. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

Future Correspondence

10. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Satya Sastri whose telephone number is 571-272-1112.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, David Wu can be reached on 571-272-1114. The fax phone numbers for the organization where this application or proceeding is assigned is (571) 273-8300 for regular communications. The unofficial direct fax phone number to the Examiner's desk is 571-273-1112.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR

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system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).



SATYA SASTRI

July 10, 2006



DAVID W. WU
SUPERVISORY PATENT EXAMINER
TECHNOLOGY CENTER 1700

PTO 06-2645

CY=JA DATE=19841012 KIND=A
PN=59-179676

METHOD FOR MANUFACTURING PRESSURE-SENSITIVE ADHESIVE TAPE
[Kan'atsusei setchaku te-pu no seizo hoho]

Takao Yoshikawa, et al.

UNITED STATES PATENT AND TRADEMARK OFFICE
Washington, D.C. February 2006

Translated by: FLS, Inc.

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| INVENTOR | (72) : | YOSHIKAWA, TAKAO; SHIBATA, YUKARI; YAMADA, SHINJI; YOSHIKAWA, SHINJI |
| APPLICANT | (71) : | NITTO ELECTRIC INDUSTRIAL CO., LTD. |
| TITLE | (54) : | METHOD FOR MANUFACTURING PRESSURE-SENSITIVE ADHESIVE TAPE |
| FOREIGN TITLE | [54A] : | KAN'ATSUSEI SETCHAKU TE-PU NO SEIZO HOHO |

1. Title of the Invention

METHOD FOR MANUFACTURING PRESSURE-SENSITIVE ADHESIVE TAPE

2. Claim(s)

(1) A method for manufacturing a pressure-sensitive tape characterized by preparing a raw material comprised mainly of 80 to 99 wt.% of a main monomer composed mainly of an acrylate or methacrylate and 1 to 20 wt.% of a copolymerizable unsaturated monomer having acidic groups, with the solvent content being 0 to 20 wt.% of and substantially free of any other medium, forming a hydrosol in which some or all of the acidic groups in the above-mentioned copolymer molecule are neutralized and also the above-mentioned copolymer having an average particle diameter of 0.01 to 0.1 μm is dispersed stably in water by adding an alkali or water thereto, and coating, on a tape support, a pressure-sensitive adhesive composition comprised by compounding a compound having two or more aziridinyl groups in the molecule as a crosslinking agent.

3. Detailed Specifications

This invention relates to a method for manufacturing an acrylic pressure-sensitive adhesive tape.

In further detail, it relates to a method for manufacturing a pressure-sensitive adhesive tape having outstanding water resistance by forming a hydrosol having a fine particle diameter in a specific range from an acrylic copolymer without using an emulsifier, and using a solvent-free-type of adhesive composition comprised by compounding an

* Number in the margin indicates pagination in the foreign text.

aziridinyl compound with this as the crosslinking agent.

As a consequence of their strong points of outstanding adhesion characteristics and durability, acrylic pressure-sensitive adhesives have become widely prevalent in recent years, replacing conventional natural rubber-based and synthetic rubber-based pressure-sensitive adhesives.

The manufacture of adhesive tapes using such acrylic pressure-sensitive adhesives in the past was generally performed in a method in which an organic solvent solution of an acrylic polymer was coated on a tape support and dried. But because a large amount of organic solvent was required in this method, various methods to replace it were proposed from the point of view of the problems of petroleum resources, preventing pollution, and so forth.

Of these, adhesive tapes using an emulsion-based pressure-sensitive adhesive free of an organic solvent has been highly regarded most recently particularly from the point of view of saving resources, being free of pollution, environmental health, etc. But, on the other hand, because an emulsifier is used, there are problems with the water resistance /462 and adhesion characteristics of the adhesive layer.

That is, a large amount (normally 8 to 10 wt.%) of an emulsifier used during the compounding of additives, such as a tackifier resin during or after polymerization, is contained in the adhesive, resulting in the water absorption being high and the water resistance being poor, from among the adhesive characteristics. Moreover, when the tape is immersed in water, it whitens within 24 hours and all of its functions are lost.

In addition, since average particle diameter of the emulsion is about 0.1 to 1 μm , the uniform film-forming capability worsens somewhat more than with an organic solvent solution type.

Meanwhile, from the point of view of film formation, as observed in the publications of Tokko No. 46-22343, Tokkai No. 50-19842, and the like, methods for manufacturing hydrosols by a so-called strippable technique, in which polymer particles having a particle diameter of 0.3 to 0.7 μm and a carboxyl group obtained in an emulsion polymerization method was stirred at high speed in the presence of an alkali, such as caustic potash, caustic soda or ammonium hydroxide, and the particle surface was scraped off to obtain fine particles having a particle diameter of 0.01 to 0.1 μm , were proposed.

However, although an improvement in the film-forming capability was confirmed on the basis of making the fine particles according to these methods, the molecular weight of the polymer that could be made into a hydrosol upon use of an emulsifier was limited; hence, it was generally difficult to make a hydrosol therefrom if the weight average molecular weight was 10^4 or higher, and only applications thereof only could be developed for the coating film and paper sizing fields primarily.

The inventors of this invention previously proposed a method in which a hydrosol, in which the below-mentioned copolymer was emulsified and dispersed as fine particles, was obtained by adding an alkali and water to a polymer having a relative high molecular weight (weight average molecular weight: 10^4 to 10^6 ; a specific copolymer having acidic groups and obtained not by polymer emulsion but by copolymerization without a solvent or in

the presence of a small amount of solvent) and neutralizing the above-mentioned acidic groups.

On the basis of the above-mentioned proposed methods, this invention was discovered as a result of painstaking investigations for the purpose of obtaining a solvent-free-type of pressure-sensitive adhesive tape having outstanding water resistance, and an aspect thereof is a method for manufacturing a pressure-sensitive tape characterized by preparing a raw material comprised mainly of 80 to 99 wt.% of a main monomer composed mainly of an acrylate or methacrylate and 1 to 20 wt.% of a copolymerizable unsaturated monomer having acidic groups, with the solvent content being 0 to 20 wt.% of and substantially free of any other medium, forming a hydrosol in which some or all of the acidic groups in the above-mentioned copolymer molecule are neutralized and also the above-mentioned copolymer having an average particle diameter of 0.01 to 0.1 μm is dispersed stably in water by adding an alkali or water thereto, and coating, on a tape support, a pressure-sensitive adhesive composition comprised by compounding a compound having two or more aziridinyl groups in the molecule as a crosslinking agent.

That is, it was discovered, in this invention, that when acrylic copolymers having acidic groups were synthesized in various polymerization methods, and also, hydrosols were formed by the aforesaid means as raw materials free of solvents or comprising a small amount of solvent, and coated on a tape support, the adhesive tapes exhibited high water-resistance characteristics because no emulsifier was contained, they exhibited outstanding film-forming properties because the particles were small,

and more remarkably improved water resistance and adhesion characteristics could be obtained than with conventional emulsion-type adhesives.

Moreover, the inventors discovered that when an aziridinyll compound was used as the crosslinking agent in particular, a satisfactory tape in which there was no whitening phenomenon, no observation of water absorption at all, remarkably outstanding water resistance, and moreover, balanced with the adhesiveness, which led them to completing this invention.

In addition, in the method of this invention, the adhesion characteristics are exceedingly homogenized since, upon obtaining hydrosol particles using an alkali and water after mixing various additives, such as a tackifier and softener compounded with a general pressure-sensitive adhesive, with the aforesaid solid matter composed mainly of an acrylic copolymer, the above-mentioned additives could be contained uniformly in each particle. Furthermore, when these additives do not adversely affect the polymerization reaction, it is possible to premix them into the system during polymerization.

Vis-à-vis, with a conventional emulsion type adhesive, each of the above-mentioned additives, and in particular, the tackifier resin, is emulsified and dispersed in an emulsion obtained by emulsion polymerization; hence, each polymer particle and additive particle were distinctive particles. And in this case, even when a uniform dispersibility is obtained, it is difficult to engineer homogenization of the adhesion characteristics, and moreover, fluctuations in the adhesion characteristics easily occur over time. /463

According to the method of this invention as such, the drawbacks

of a pressure-sensitive adhesive tape using a conventional emulsion-type adhesive can be avoided altogether, and the water resistance and adhesion characteristics can be improved. Moreover, since the hydrosol consists of fine particles, during manufacture of the tape, a coating film provided with the same outstanding performance as one when an organic solvent solution was used is obtained. And the coating workability is satisfactory, the solid content concentration is relatively high at about 25 to 45 wt.%, and a coating work in which the thickness is even is possible even by the use of equipment for coating work commonly used in the past.

In this invention, any conventionally-known method, such as a bulk polymerization method, solution polymerization method, emulsion polymerization method or pearl polymerization method, can be adopted while synthesizing the acrylic copolymer comprising 80 to 99 wt.% of a main monomer composed mainly of an acrylate or methacrylate and 1 to 20 wt.% of a copolymerizable unsaturated monomer having acidic groups. But a bulk polymerization method or a solution polymerization method is especially ideal with the use of a small amount of solvent.

The acrylic copolymer obtained thereby is used as is in the bulk polymerization method, from out of these polymerization methods as a raw material for making the hydrosol.

In the solution polymerization method, the organic solvent is removed after synthesis by a means, such as distillation, when the amount is greater than 20 wt.% of the amount of the solvent used, but even if the amount is 20 wt.% or less, it is desirable to reduce or eliminate the solvent content by the above-mentioned means.

Various general organic solvents can be used for the solvent used in this solution polymerization method. But preferably, it is desirable to use an alcohol-based hydrophilic medium, such as methanol, ethanol, n-butanol, n-propanol, isopropyl alcohol or sec-butanol, or an oligomer or prepolymer having a hydrophobic group, such as a hydroxyl group, carboxyl group and amino group.

Meanwhile, a solid matter substantially free of a medium is obtained by removing the medium used respectively in other polymerization methods by a suitable means after the synthesis. That is, moisture can be removed by coagulating and dispersing it by salting out in, e.g., an emulsion polymerization method, and further, the moisture is removed by filtering out the granular polymer in a pearl polymerization method. Although it is desirable to completely remove the medium other than the organic solvent at this time, a residue thereof is allowed more or less.

Moreover, in the emulsion polymerization and pearl polymerization methods, a part of the emulsifier used during polymerization adheres to the polymer particle surface, but almost all of this emulsifier is removed during the above-mentioned medium removal operation, and moreover, by washing, if needed.

In addition, in the emulsion polymerization method, a three-dimensional copolymer obtained to a high degree can be produced sometimes, but such a three-dimensional polymer becomes a hindrance for making a hydrosol in the succeeding steps; hence, it is desirable to prevent formation of such as polymer as much as possible during emulsion polymerization. Supposing a part thereof is formed, it is desirable to masticate it under

conditions in which a high shear force is exerted by using a mixing roll, Banbury mixer, etc.

The main monomer used for obtaining the acrylic copolymer is composed mainly of an alkyl ester of acrylic acid or methacrylic acid having an alkyl group with 2 to 15 carbons, such as ethyl acrylate, butyl acrylate, 2-ethylhexyl acrylate, isooctyl acrylate, ethyl methacrylate and butyl methacrylate, and another monomer copolymerizable with it can be combined therewith.

In addition to an alkyl ester of acrylic acid or methacrylic acid having an alkyl group in which the number of carbons are outside the aforesaid range, such as methyl acrylate or methyl methacrylate; and vinyl acetate, acrylonitrile, styrene, 2-methoxyethyl acrylate, vinyl ether, and the like, various functional monomers, such as glycidyl acrylate, glycidyl methacrylate, hydroxyethyl methacrylate, acrylamides and methylolacrylamides, are included extensively for the above-mentioned other monomer.

An unsaturated carboxylic acid having a carboxyl group as the acidic group, such as acrylic acid, methacrylic acid, crotonic acid, itaconic acid, maleic acid and fumaric acid; an unsaturated sulfonic acid having a sulfonic group as the acidic group, such as styrene sulfonic acid, /464 allyl sulfonic acid, sulfopropyl acrylate, 2-acryloyloxynaphthalene-2-sulfonic acid, 2-methacryloyloxynaphthalene-2-sulfonic acid, 2-acrylamide-2-methylpropane sulfonic acid and 2-acryloyloxybenzene sulfonic acid; and the like can be cited as examples of the copolymerizable

unsaturated monomer having an acidic group used with the above-mentioned main monomer, which moreover, can have another acidic group. And one or more of these compounds may be used.

It is necessary that the proportion of the main monomer used be 80 to 99 wt.% and that of the copolymerizable unsaturated monomer having an acidic group be 1 to 20 wt.%. In particular, the proportion of the former used should be 90 to 97 wt.% and that of the latter should be 3 to 10 wt.%. It is, of course, unsuitable when the copolymerizable unsaturated monomer having an acidic group is less than 1 wt.% because it is difficult to make the hydrosol or if it is greater than 20 wt.% because a reduction in the adhesion characteristics, such as the tackiness, are induced.

It is desirable that the molecular weight of the acrylic copolymer synthesized at the above-mentioned proportions of use generally range from 10^4 to 10^6 . If the molecular weight is too low, the cohesive power of the adhesive tape becomes poor and a crosslinking agent is required for improving this, but in this case, there is a problem because the stability of the hydrosol is compromised. Moreover, if the molecular weight is much too high, making a hydrosol by using an alkali and water becomes difficult, which is not desirable from the standpoint of the tacky adhesion characteristics.

In this invention, by adding an alkali and water to the raw material composed mainly of an acrylic copolymer prepared as mentioned above (as previously stated, various additives have been included, as needed) and stirring and mixing this, a portion or all of the acidic groups in the

copolymer are neutralized, on the one hand, and a hydrosol is produced in which the above-mentioned copolymer is dispersed stably in water.

An alkali that can easily disperse while coating the hydrosol on a support and subsequently heating and drying it can be chosen for the alkali used here. By using such an alkali, the adverse effects of the mixing of the alkali on the adhesion characteristics can be prevented, thus satisfactory adhesion characteristics can be obtained.

Ammonia is a typical dispersible alkali. Otherwise, α -aminoethyl alcohol, ethylamine, propylamine, and the like also can be used. The amount of the alkali used normally may be about 0.1 to 2 equivalents with respect to the acidic group contained in the acrylic copolymer.

The temperature during the neutralizing treatment is maintained at a constant temperature depending on the type, attributes, and the like, of acrylic copolymer, but generally it is 30 to 80°C. The method for neutralizing and producing a hydrosol can be performed in a method in which a prescribed amount of alkali and water are added at once then stirred and mixed well. After adding a prescribed amount of alkali or alkali aqueous solution and stirring and mixing it well, an O/W-type dispersion in which the water becomes a continuous phase and the copolymer particles are dispersed therein can be produced by gradually adding water to cause a phase transition.

The dispersion thus obtained is a hydrosol in which copolymer particles, which are dispersed particles, are dispersed homogeneously and stably in water. The viscosity thereof at 25°C is generally 30 to 1,000 poise, and the solid content concentration is 25 to 45 wt.%.

Moreover, additives, such as tackifiers, softeners, plasticizers, fillers and colorants, can be added to the solid raw material before making the hydrosol, as previously stated, during manufacture of this hydrosol. In this case, normally these are added after polymerization, but as the circumstances permit, they can be added before polymerization. Of course, each of the above-mentioned additives can be added to the sol after making the hydrosol.

1,1'-(methylene-di-P-phenylene)bis-3,3-aziridinyl urea,
1,1'-(hexamethylene)bis-3,3-aziridinyl urea, ethylene
bis(2-aziridinylpropionate), tris(1-aziridinyl)phosphineoxide, 2,4,6-
triaziridinyl-1,3,5-triazine, 2,2-bis(hydroxymethyl)butanol- tris[3- /465
(1-aziridinyl)propionate], and the like are cited as examples of the compound
having two or more aziridinyl group in the molecule and added to the hydrosol
obtained by the above process. One or more of these can be mixed and
used.

The amount of this aziridinyl compound used should be 0.01 to 5 parts by weight per 100 parts by weight of the acrylic copolymer in the hydrosol. It is not preferable if the amount used is too low because the water resistance of the pressure-sensitive adhesive tape decreases or if it is too high because the adhesiveness decreases.

The above-mentioned hydrosol can be coated to an even thickness on one or both surfaces of various types of tape supports, such as plastic film, nonwoven fabric, woven fabric, paper and foil with commonly-used coating equipment, and the moisture is eliminated by heating and drying after this coating step to disperse the dispersible alkali; hence, a

pressure-sensitive adhesive tape having satisfactory the target water resistance and adhesion characteristics can be obtained.

This invention will now be described more specifically, but it is not restricted to these practical examples. Moreover, 'part(s)' in the document hereafter wholly means 'part(s) by weight.'

In addition, evaluation of the pressure-sensitive adhesive tape obtained in the present invention was performed according to the following methods.

<Bond strength>

A 20 mm wide, 150 mm long pressure-sensitive adhesive tape was prepared, and peeled 180 degrees in accordance with JIS Z-1528 to measure the bond strength (g/20mm).

<Holding power>

A 10 mm wide, 150 mm long pressure-sensitive adhesive tape was prepared and a 500 g load was applied at 40°C to a 10 mm wide, 20 mm long piece after sticking it to a bakelite plate to measure the time (minutes) until the load fell.

<Water resistance>

The change over time (in days) after immersing the pressure-sensitive adhesive tape in 20°C water for 7 days was examined. An evaluation was given at a (O) rank when a whitening phenomenon was not confirmed after 7 days, at a (Δ) rank when a slight whitening phenomenon was confirmed after 3 days, and at a (x) rank when a marked whitening phenomenon was confirmed after 1 day.

<Water absorption>

The pressure-sensitive adhesive was coated on one side of a 25 μm thick polyester film to a dried thickness of 50 μm , and after drying for 8 minutes at 100°C, it was immersed in water, withdrawn 7 days later, and the weight thereof was measured after removing the water droplets on the surface to find the water absorption (%).

Practical Example 1

A 1L four-necked flask was charged with 10 g of a composition comprising 80 g 2-ethylhexyl acrylate, 20 g ethyl acrylate, 5 g acrylic acid, 5 g 2-hydroxyethyl acrylate, 0.15 g azobisisobutyronitrile, and 0.05 g lauryl mercaptan, and displaced with nitrogen for 40 minutes under stirring. Next, this was allowed to react for 4 hours at 88°C while adding the balance of the composition dropwise through a dropping funnel, and a copolymer having a weight average molecular weight of 6×10^5 according to GPC was synthesized.

Next, 0.45 equivalents of ammonia with respect to the carboxyl group of this copolymer and 200 g water were added, a neutralization treatment was performed at a temperature of 69°C to cause a phase transition, and a hydrosol in which the continuous phase was water was obtained. The hydrosol obtained by the above process has a viscosity at 25°C of 120 poise, a solid content concentration of 32.5 wt.% and an average particle diameter of 0.07 μm according to a nanosizer.

A crosslinking agent solution comprised by dissolving 0.5 parts 1,1'-(methylene-di-P-phenylene)bis-3,3'-aziridinyl urea in 5 parts dimethylformamide was compounded with 100 parts of the above-mentioned

hydrosol polymer solid content to obtain a pressure-sensitive adhesive composition.

This composition was coated on a 50 μm thick polyester film and heated and dried for 4 minutes at 130°C to obtain a pressure-sensitive adhesive tape having a 50 μm thick paste thickness.

Comparative Example 1

1.2 parts of triglycidylisocyanurate with respect to 100 parts of polymer solid content were added as the crosslinking agent to the hydrosol obtained in Practical Example 1, and this adhesive composition was used to obtain a pressure-sensitive adhesive tape as in Practical Example 1.

Comparative Example 2

2.2 parts tributoxymethylmelamine with respect to 100 parts of the polymer solid content were added to the hydrosol obtained in /466 Practical Example 1 as a crosslinking agent, and this adhesive composition was used to obtain a pressure-sensitive adhesive tape as in Practical Example 1.

Comparative Example 3

The monomer mixture having the same composition as in Practical Example 1 was subjected to an emulsion polymerization by using 2 parts sodium laurylsulfate as an emulsifier, thioglycol acid as a chain transfer agent, and potassium persulfate as a polymerization initiator, and a polymer emulsion having a weight average molecular weight of 5×10^5 and an average particle diameter of 0.28 μm was obtained. The same crosslinking agent as in Practical Example 1 was compounded with this emulsion and a pressure-sensitive adhesive tape was obtained as in Practical Example

1 using this adhesive composition.

The results of evaluating the respective pressure-sensitive adhesive tapes in Practical Example 1 and Comparative Examples 1 to 3 above are as shown in Table 1 next.

Table 1

| | Bond Strength (g/20 mm wide) | Holding Power (min.) | Water Resistance | Water Absorption (%) |
|-----------------------|---------------------------------|-------------------------|---------------------|-------------------------|
| Practical Example 1 | 820 | ≥1,000 | 0 | 0.9 |
| Comparative Example 1 | 700 | ≥1,000 | Δ | 13.2 |
| Comparative Example 2 | 620 | 800 | Δ | 15.5 |
| Comparative Example 3 | 670 | 1,000 | Δ | 53.2 |

Practical Example 2

Except for changing the amount of acrylic acid used to 20 g, a hydrosol and pressure-sensitive adhesive tape were obtained as in Practical Example 1.

Comparative Example 5

Except for changing the amount of acrylic acid used to 1 g, a pressure-sensitive adhesive tape was obtained as in Practical Example 1, but a hydrosol having a microscopic particle size was not formed even by neutralization with ammonia. Instead, it was an aqueous dispersion having a large particle size and exceedingly poor stability. A coatable adhesive could not be obtained.

Practical Example 3

A 1L four-necked flask was charged with 10 g of a monomer mixture having a composition comprising 80 g n-butyl acrylate, 20 g ethyl acrylate, 10 g methacrylic acid, 0.1 g benzoyl peroxide and 5 g sec-butanol, and 5 g sec-butanol, and displaced with nitrogen for 40 minutes at 40°C under stirring. 0.1 g benzoyl peroxide was added after that and completely

dissolved, after which the temperature was raised to 80°C. Then, the balance of the monomer mixture was added dropwise over 2 hours at a speed of about 0.87 g/min through a dropping funnel, allowed to react for 4 hours at 85±5°C, and a copolymer having a weight average molecular weight of 3×10^5 according to GPC was synthesized.

0.25 equivalents of ammonia with respect to the carboxyl groups of this copolymer and 170 parts water with respect to 100 weight parts of copolymer were neutralized at a temperature of 50±3°C to cause a phase transition, and a hydrosol having a continuous phase of water was obtained.

The hydrosol obtained by the above process had a viscosity at 25°C of 520 poise, a solid content concentration of 33 wt.% and an average particle diameter of 0.05 μm .

0.2 parts of 2,2-bishydroxymethylbutanol-tris[3-(1-aziridinyl) propionate] were compounded with 100 parts of the polymer of the above hydrosol, and a pressure-sensitive adhesive composition was prepared. A pressure-sensitive adhesive tape was obtained from this composition as in Practical Example 1.

Practical Example 4

| | |
|------------------------|--------|
| 2-ethylhexyl acrylate | 60 g |
| Ethyl acrylate | 40 g |
| Methacrylic acid | 15 g |
| Azobisisobutyronitrile | 0.15 g |
| Toluene | 200 g |

A 1L flask was charged with the above-mentioned composition, allowed to react for 4 hours at 65°C under stirring, and a copolymer having a weight average molecular weight of 7×10^5 according to GPC was synthesized. The toluene was distilled from this polymerization reaction product by

reduced-pressure distillation.

Then 250 parts of water were added to 1/8 equivalents of aqueous ammonia with respect to the carboxyl groups of this copolymer to 100 parts of the copolymer, neutralized to cause a phase transition, and a hydrosol having a continuous phase of water was obtained. A pressure-/467
sensitive adhesive tape was obtained from this hydrosol as in Practical Example 3.

Practical Example 5

| | |
|------------------------------------|--------|
| N-octyl acrylate | 65 g |
| Methyl methacrylate | 35 g |
| Methacrylic acid | 10 g |
| Lauryl mercaptan | 0.15 g |
| Polyoxyethylene alkyl phenol ether | 3 g |
| Water | 160 g |

A 1L flask was charged with the above-mentioned composition, heated to 70°C under stirring, a polymerization initiator in which 0.1 g potassium persulfate was added to 5 g of water was added thereafter, allowed to react for 3 hours at 70°C, and a copolymer emulsion having a weight average molecular weight of 1.5×10^5 according to GPC was synthesized. This copolymer emulsion was salted out, the resulting precipitated copolymer was filtered, washed with water and dried.

A hydrosol was obtained next by causing a phase transition by performing the neutralization treatment as in Practical Example 1.

The pressure-sensitive adhesive tape was obtained as in Practical Example 1 hereafter while using this hydrosol.

Practical Example 6

| | |
|------------------|-------|
| N-butyl acrylate | 65 g |
| Ethyl acrylate | 35 g |
| Acrylic acid | 7 g |
| Benzoyl peroxide | 0.1 g |

A 1L flask was charged with 280 g of a 3×10^{-4} wt.% polyvinyl alcohol aqueous solution, the above-mentioned composition was added and allowed to react for 4 hours at 70°C under stirring, and pearl particles having an average particle diameter of 1.5 mm were obtained as a copolymer having a weight average molecular weight of 6×10^5 according to GPC. These particles were filtered, washed with water and dried.

Next, 15 parts of n-propyl alcohol were added to 100 parts of this copolymer, sufficiently dissolved, and a hydrosol was subsequently obtained as in Practical Example 3. By using this hydrosol, a pressure-sensitive adhesive tape was obtained as in Practical Example 3.

The results of evaluating the performance of each of the pressure-sensitive adhesive tapes in Practical Examples 2 to 6 above are as shown in Table 2.

Table 2

| | Bond Strength (g/20 mm wide) | Holding Power (min.) | Water Resistance | Water Absorption (%) | Average Particle Diameter of Hydrosol (μ m) |
|------------------------|---------------------------------|----------------------------|---------------------|----------------------------|--|
| Practical Example 2 | 720 | $\geq 1,000$ | ○ | 1.5 | 0.07 |
| Practical Example 3 | 870 | $\geq 1,000$ | ○ | 1.2 | 0.06 |
| Practical Example 4 | 650 | $\geq 1,000$ | ○ | 1.0 | 0.04 |
| Practical Example 5 | 300 | $\geq 1,000$ | ○ | 7.4 | 0.08 |
| Practical Example 6 | 760 | $\geq 1,000$ | ○ | 4.5 | 0.07 |

As evident from the above results, it is seen that a pressure-sensitive adhesive tape obtained in the method of manufacture of this invention has balanced bond strength and holding power, exhibits outstanding water resistance without absorbing water or whitening at all, and there are no changes in this water resistance over time.